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(71) Applicant: PROPPER MANUFACTURING COMPANY, INC. [US/US]; 36-04 Skillman Avenue, Long Island City, NY 11101 (US).

(72) Inventor: PLATKO, Frank, E.; 94 Village Drive, Shelton, CT 06484 (US).

(74) Agents: LARSON, Marina, T. et al.; Oppedahl & Larson, Suite 309, 1992 Commerce Street, Yorktown Heights, NY 10598-4412 (US).

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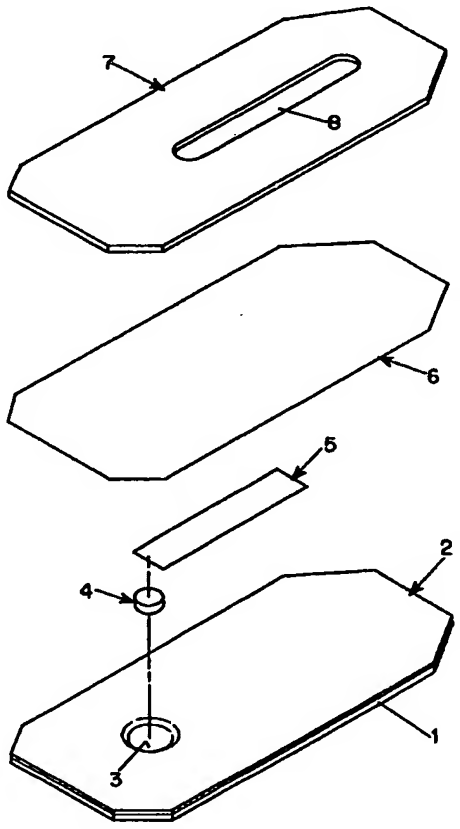
(54) Title: STEAM STERILIZATION INDICATOR

(57) Abstract

A steam sterilization indicator comprises: (a) a foil backing (1) having a thickness of at least 5 mil; (b) a wick (5) disposed on the foil backing (1); (c) a meltable chemical (4) disposed on the foil backing (1) in a position relative to the wick (5) such that upon melting the melted chemical will be transported through the wick (5), said meltable chemical (4) being melted at or below a target sterilization temperature in the presence of saturated steam, and at a temperature above the target sterilization temperature in the abscence of saturated steam; (d) a steam permeable cover (6) disposed over wick (5) and the meltable chemical (4); and (e) an aggressive high temperature silicone adhesive (2), said adhesive (2) adhering the foil backing (1) to the steam permeable cover (6) whereby a substantially sealed envelope surrounding the wick (5) and the meltable chemical (4) is formed from the foil backing (1), the steam permeable cover (6) and the adhesive (2). A suitable adhesive for use in this device is the type of adhesive employed in Densil® 2078, a 2 mil silicone adhesive film.



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STEAM STERILIZATION INDICATOR

BACKGROUND OF THE INVENTION

This application relates to an indicator for use in monitoring the effectiveness of a steam sterilization process. More specifically, the application relates to a novel steam sterilization indicator which integrates the effects of time and temperature and saturated steam.

Steam sterilization integrators have been described in U.S. Patents Nos. 3,324,723; 3,981,683; 4,448,548 and 5,158,363 each of which is incorporated herein be reference. In general, an integrator device is formed of an envelope which encloses a wick and meltable chemical.

The meltable chemical is selected from among chemicals having a normal melting point above a target sterilization temperature which is depressed to below the target sterilization temperature when contacted with saturated steam. When the indicator is exposed to saturated steam at a sufficiently high temperature, the chemical melts and travels along the wick, thus providing a visual indication of the time period over which conditions of sufficient temperature and steam exposure are maintained.

The envelope is selected to fulfill two functions: to prevent leakage of the chemical from the indicator, and to control the rate at which steam from the sterilizer can contact the meltable chemical. As is apparent from U.S. 3,981,683, 4,448,548 and 5,158,363 and various commercial products sold under the trademarks SteriGage®, SteamLine®, Vapor Line™ and Therma-Log®, a common approach to the formation of the envelope is two utilize a foil backing to which a steam permeable cover is glued.

In seeking an ideal adhesive for use in this application, the present inventor has formulated criteria that the adhesive should meet. First, it must be stable at the temperatures which occur during sterilization, i.e., approximately 121 to 134°C so that the indicator

does not simply fall apart. Second, it must form a good bond between the foil backing and the cover to minimize steam leakage through gaps in the adhesive, which could negate the rate controlling effect of the cover. Finally, it must be able to maintain adhesion through the torsional stresses on the indicator which result from the vacuum and steam pulses of the sterilizer. It is the object of this invention to provide a steam sterilization indicator having an adhesive which meets these requirements.

SUMMARY OF THE INVENTION

This and other objectives are achieved by a steam sterilization indicator in accordance with the invention comprising:

(a) a foil backing having a thickness of at least 5 mil;

(b) a wick disposed on the foil backing;

(c) a meltable chemical disposed on the foil backing in a position relative to the wick such that upon melting the melted chemical will be transported through the wick, said meltable chemical being melted at or below a target sterilization temperature in the presence of saturated steam, and at a temperature above the target sterilization temperature in the absence of saturated steam;

(d) a steam permeable cover disposed over wick and the meltable chemical; and

(e) an aggressive high temperature silicone adhesive, said adhesive adhering the foil backing to the steam permeable cover whereby a substantially sealed envelope surrounding the wick and the meltable chemical is formed from the foil backing the steam permeable cover and the adhesive. A preferred adhesive for use in this device is the type of adhesive employed in Densil® 2078, a 2 mil silicone adhesive film.

BRIEF DESCRIPTION OF THE DRAWING

Fig. 1 shows an exploded view of an indicator in accordance with the invention.

DETAILED DESCRIPTION OF THE INVENTION

Fig. 1 shows an exploded view of an indicator in accordance with the invention. The indicator is formed from a metal foil backing 1 to which is applied a layer of a high performance silicone adhesive 2. Preferably, an embossment 3 is formed in the foil backing 1 to receive a tablet of meltable chemical 4. Wick 5 is disposed on the foil backing 1 in contact with or in close proximity to the tablet of meltable chemical 4. A steam permeable cover 6 is placed over the wick 5 and the tablet of meltable chemical 4. The steam permeable cover 6, the adhesive layer 2 and the foil backing 1 taken together form a substantially sealed envelope surrounding wick 5 and the tablet of meltable chemical 4. Finally, an optional paper cover plate 7 having a window 8 therein is affixed over the steam permeable cover 6.

A suitable metal foil for use as the foil backing 1 is 5 mil aluminum foil Grade: 1100-0 MF, "A" wettable available from Metal Foils, Inc., Willoughby, Ohio. Other metal foils of comparable thickness and rigidity may also be employed provided they are sufficiently malleable to permit the formation of the embossment 3.

The meltable chemical 4 may be in the form of a regularly shaped tablet or pellet as shown in Fig. 1, or in the form of an irregularly-shaped, amorphous mass. In general, the tablet may by any chemical which melts at or below a target sterilization temperature in the presence of saturated steam, and at a temperature above the target sterilization temperature in the absence of saturated steam. A preferred chemical for use in steam sterilizers having a target temperature of 121-134°C is salicylamide. A binder may be added to the tablets as described in U.S. Patent No. 4,448,548, but is not required.

When the meltable chemical selected is itself colored, no additional materials are required within the tablet 4. In the case of colorless or substantially colorless compounds, a dye may be added to facilitate easy observation of the advance of the meltable chemical along the wick. Suitable dyes include Orasol Brilliant Blue, Spirit Soluble Fast Black (also called Zapon Black X-50), and Spirit Soluble Orange RR.

The wick is advantageously a strip of filter paper through which the melted chemical will migrate. The specific grade of filter paper is selected to yield a device having an appropriate migration distance for the melted chemical at the sterilization temperature in the normal sterilization cycle time. Specific suitable papers include Whatman No. 1 Chrome and Ahlstrom Grade 602-025.

The steam permeable cover may be made from various materials which are permeable to steam at the target sterilization temperature. A preferred material for use as the steam permeable cover is 1.2 mil transverse oriented polypropylene. In general, films of about 0.5 to 2 mil can be used depending on the degree of steam transmission required.

The optional paper cover 7 is applied to facilitate marking of the indicator with instructions, a line to indicate a positive or negative test result and indications of origin. It should be substantially lint free and capable of being printed. The paper cover is attached to the outer surface of the steam permeable cover by an adhesive such as a pressure sensitive latex emulsion. Since the performance of the indicator has been observed to be the same whether or not the paper cover is included, however, the specific identities of the paper and the adhesive holding it to the steam permeable cover are not critical.

The key element in the present invention is the use of a high performance silicone adhesive such as the

adhesives used in Densil® 2078 (available from Flexcon Corp., Spencer, Massachusetts), or CW-14HT (available from Specialty Tapes, a Division of RSW, Inc., Racine, Wisconsin), and the use of this adhesive in combination with the 5 mil or greater foil backing. The silicone adhesive is preferably applied as a film to either the foil backing or the steam permeable cover. In the case of Densil® 2078, this film to 2 mil thick. Thinner films, for example the 1 mil film sold as Densil® 1078 can also be employed, as can thicker films, although in the latter case the cost may be prohibitive.

The adhesive bonds the steam permeable cover to the foil backing to form a substantially sealed envelope around the wick and the tablet of meltable chemical. As used herein, the term "substantially sealed" means that a seal is formed which is free of distinct channels or holes through which gases, including steam, may pass in an unobstructed manner. It will be understood, however, that the adhesive may possess some degree of permeability to steam or other gases, which may be greater than, equal to or lesser than the permeability of the steam permeable cover, and thus that some portion of the steam probably enters the device through the adhesive rather than through the steam permeable film.

The combination of the high performance silicone adhesive and the thicker foil backing makes it possible to achieve an indicator of small size (an important feature for minimizing material costs) that has excellent performance characteristics, including a low failure rate, high indicator-to-indicator reproducibility, and low indicator distortion resulting in improved indicator readability. This achievement of a highly successful indicator of small size using this combination of materials is surprising in view of the knowledge available in the prior art. U.S. Patent No. 3,981,683 discloses an indicator in which a silicone adhesive is used, but the indicators made using this technology are of larger size,

i.e., about 100 mm by 21 mm. U.S. Patent No. 4,448,548 discloses smaller size indicators, but suggests that silicone adhesives are not suitable for such devices. Indeed, U.S. Patent No. 4,448,548 uses Densil® 2078 and a 3 mil foil backing as a comparative example and suggests that the performance is defective. The '548 patent hypothesizes that this may be due to the formation of a seal by the acrylic adhesive which is better than the seal achieved by the silicone adhesive.

Although nowhere reflected in the prior art, applicant's experience has been that indicators made using Densil 2078® adhesive films and 3 mil foil, i.e., an indicator made in accordance with the '548 patent, undergo significant physical twisting and deformation during a sterilization cycle. While not intending to be bound by any specific theory, this deformation may result in the formation of holes through which non-selective transport of gases, including steam, is possible. This would result in an increase in the exposure of the interior of the indicator to steam, and thus in the potential for false positive results.

Contrary to the '548 patents hypothesis of poor adhesion by the silicone, it is applicant's belief that this deformation occurs because the high performance adhesive is actually too good an adhesive, and fails to allow for the stresses induced by the vacuum and steam pulses of the sterilizer. The use of a thicker foil backing overcomes this problem, which is wholly unrecognized in the art, and produces a useful indicator of very good performance characteristics.

The invention will now be further described by way of the following non-limiting examples.

EXAMPLE 1

Sterilization indicators were prepared in the manner depicted in Fig. 1 using 5 mil aluminum foil Grade: 1100-0 MF, "A" wettable as the foil backing, a tablet

consisting of salicylamide and a dye, an Ahlstrom 602-025 paper wick, Densil® 2078 laminating adhesive, a 1.2 mil polypropylene film and a paper cover. The indicators were run in several batches in a standard QC cycle in a pre-vacuum sterilizer at 272°F for varying periods of time, and the distance that the melted chemical migrated in the wick was measured. The results are summarized in Table 1.

TABLE 1

SAMPLE	SAMPLE SIZE	EXPOSURE TIME	TEMP (°F)	Length of Migration (mm)		
				HIGH	LOW	AVG
1	8	45 sec	272	9	8	8.5
2A	2	1.5 min	272	14	14	14
2B	5	1.5 min	272	14	12	13
3A	2	2 min	272	16	16	16
3B	5	2 min	272	15.5	15.5	15.5
4	8	4 min	272	29	28	28.8

EXAMPLE 2

Indicators of the same type used in Example 1 were tested in a gravity-type sterilizer operated at 250°F. The results are summarized in Table 2. In Table 2, the notation "max" reflects that the chemical migrated the full length of the indicator strip.

TABLE 2

SAMPLE	SAMPLE SIZE	EXPOSURE TIME	TEMP (°F)	Length of Migration (mm)		
				HIGH	LOW	AVG
5	8	5 min	250	13	10	10.5
6A	2	10 min	250	20	20	20
6B	4	10 min	250	19	17	18.3
6C	5	10 min	250	22	20	21
7	8	20 min	250	max	max	max
8	8	30 min	250	max	max	max

Example 3

Indicators were made as described in Example 1, except that 1.0 mil polypropylene film was used. The indicators were tested at 250 and 272°F. The results are summarized in Table 3 as samples 9, 10 and 11 together with results for 1.2 mil film for comparison.

TABLE 3

SAMPLE	SAMPLE SIZE	EXPOSURE TIME	TEMP (°F)	Length of Migration (mm)		
				HIGH	LOW	AVG
9	8	45 sec	272	11	10	10.5
1	8	45 sec	272	9	8	8.5
10	8	4 min	272	max (32)	29	31
4	8	4 min	272	29	28	28.8
11	7	5 min	250	13	10	11.4
5	8	5 min	250	13	10	10.5

In the case of sample 11, one indicator was excluded from the results in the table because of uncharacteristic behavior. This indicator migrated 23 mm. Upon microscopic examination of the indicator, however, a crease was observed in the foil. It is believed that this crease caused a leakage of steam and that this resulted in the unusually long migration distance.

Comparative Example

Indicators were made using 3 mil foil and 1.0 mil or 1.2 mil polypropylene film and tested at 250 or 270°F. All samples were tested in the same sterilizer used in the preceding three examples, except samples 13 and 15 which were run in an Amsco Eagle sterilizer. The results are summarized in Table 4.

TABLE 4

SAMPLE	SAMPLE SIZE	EXPOSURE TIME	TEMP (°F)	Length of Migration (mm)		
				HIGH	LOW	AVG
12 A (1 mil)	4	45 sec	272	19	17	18
12 B (1 mil)	4	45 sec	272	15	13	13.8
13 (1 mil)	5	1 min	272	21	15	14.6
14 A (1 mil)	4	4 min	272	max	max	max
14 B (1 mil)	12	4 min	272	max	24	29
15 (1 mil)	4	5 min	250	20	9	12.8
16 (1 mil)	4	20 min	250	max	20	24
17 (1 mil)	4	20 min	250	18	12	14.5
18 (1.2 mil)	4	20 min	250	10	8	9

These results show that the indicators using the three mil foils were erratic in the length of migration observed. Such erratic behavior is unacceptable in a steam sterilization where the migration distance is compared to a standard mark to determine whether to pass or fail the sterilizer contents.

During the testing of the indicators using the 3 mil foil, some physical distortion of the indicators was observed in most instances. This distortion sometimes caused lifting of the steam permeable cover from the foil, and made the indicator prone to leakage of the steam into the indicator (which could cause erratic movement of the chemical and dye) and leakage of composite out of the indicator. In contrast, little or

no distortion of the indicators with a 5 mil foil backing was observed.

EXAMPLE 5

A side by side comparison was performed between indicators made using 3 mil foil backing and 5 mil foil backing. The test was run in an Amsco Eagle sterilizer operating at 272°F with an exposure time of 1.5 minutes. As shown in Table 5, in this particular test, any differences in the performance of the two types of indicators was minor. Nevertheless, based on the overall pattern of behavior of the 3 mil indicators, it is believed that this was an artifact of the small sample size rather than a true indication of the comparative reliability of the indicators.

TABLE 5

SAMPLE	SAMPLE SIZE	EXPOSURE TIME	TEMP (°F)	Length of Migration (mm)		
				HIGH	LOW	AVG
19 (3 mil)	12	1.5 min	272	15	12	13.5
20 (5 mil)	12	1.5 min	272	15	14	14.3

I Claim:

1. A steam sterilization indicator comprising:

(a) a foil backing (1) having a thickness of at least 5 mil;

(b) a wick (5) disposed on the foil backing (1);

(c) a meltable chemical (4) disposed on the foil backing (1) in a position relative to the wick (5) such that upon melting the melted chemical (4) will be transported through the wick (5), said meltable chemical (4) being melted at or below a target sterilization temperature in the presence of saturated steam, and at a temperature above the target sterilization temperature in the absence of saturated steam;

(d) a steam permeable cover (6) disposed over wick (5) and the meltable chemical (4); and

(e) an aggressive high temperature silicone adhesive (2), said adhesive adhering the foil backing (1) to the steam permeable cover (6) whereby a substantially sealed envelope surrounding the wick (5) and the meltable chemical (4) is formed from the foil backing (1), the steam permeable cover (6) and the adhesive (2).

2. A steam sterilization indicator according to claim 1, wherein the adhesive (2) is the adhesive employed in Densil® 2078.

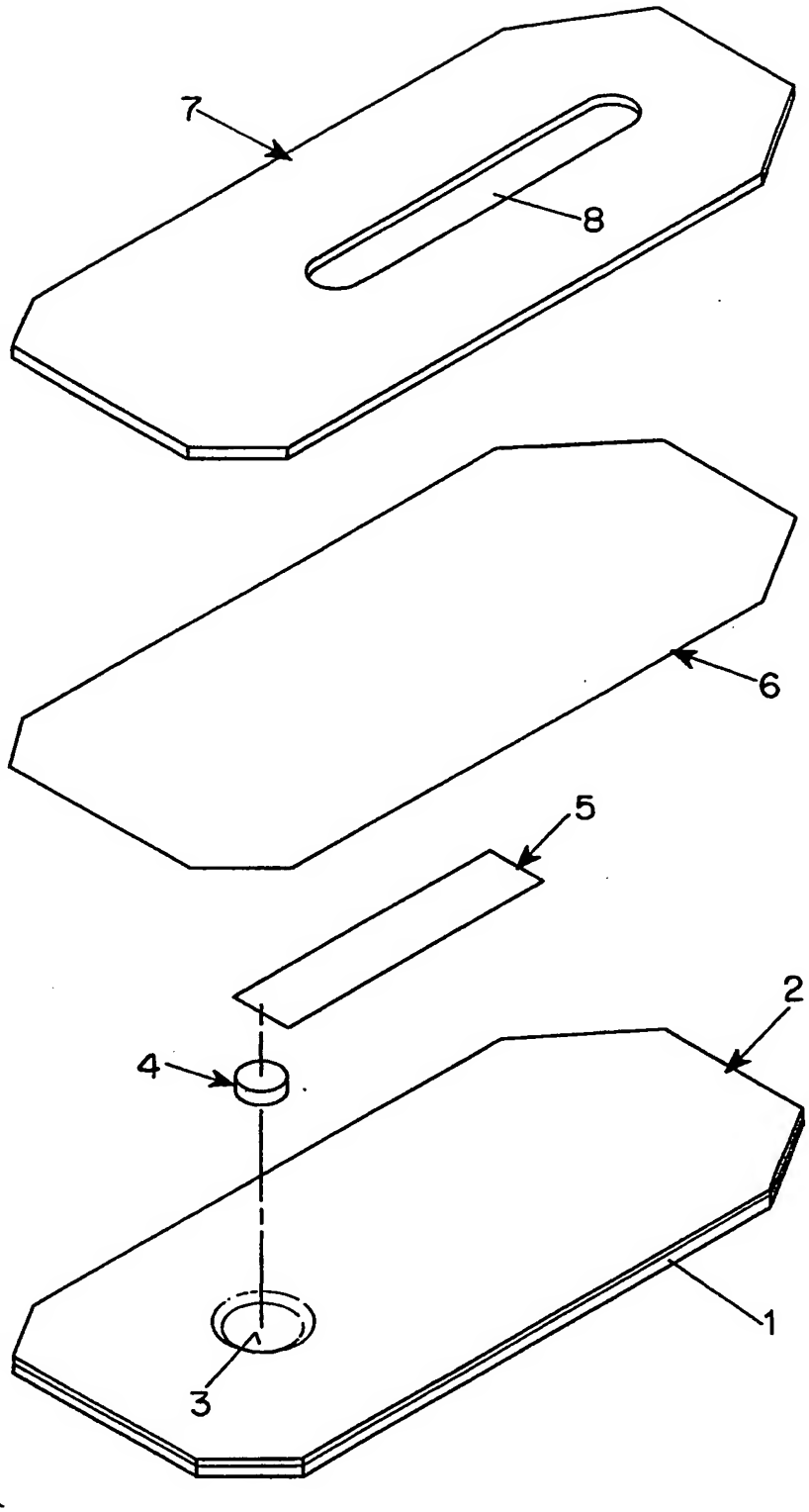
3. A steam sterilization indicator according to claim 1 or 2, wherein the adhesive (2) is applied to the foil backing (1) as a film.

4. A steam sterilization indicator according to claim 1 or 2, wherein the adhesive (2) is applied to the steam permeable cover layer (6) as a film.

5. A steam sterilization indicator according to any of claims 1-4, wherein the meltable chemical (4) comprises salicylamide and a dye.

6. A steam sterilization indicator according to any of claims 1-5, wherein the steam permeable film (6) is polypropylene.

FIG. 1



INTERNATIONAL SEARCH REPORT

International application No.
PCT/US95/02341

A. CLASSIFICATION OF SUBJECT MATTER

IPC(6) :G01K 11/06; G01N 31/22
US CL :422/57, 58; 374/106, 160; 116/219
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 422/56, 57, 58; 374/102, 106, 160, 162; 116/207, 217, 219; 435/31; 436/1

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

NONE

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

NONE

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US, A, 3,981,683 (LARSSON ET AL.) 21 SEPTEMBER 1976. SEE ENTIRE DOCUMENT.	1
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Y		2, 3, 4
Y	US, A, 4,448,548 (FOLEY) 15 MAY 1984. SEE ENTIRE DOCUMENT, PARTICULARLY COLUMN 7, LINES 14-21.	2, 3, 4
A	US, A, 5,158,363 (SPEELMAN ET AL.) 27 OCTOBER 1992.	
A	US, A, 4,138,216 (LARSSON ET AL.) 06 FEBRUARY 1979	
A	US, A, 3,324,723 (RITCHIE ET AL.) 13 JUNE 1967.	
A	US, A, 3,954,011 (MANSKE) 04 MAY 1976.	

[X] Further documents are listed in the continuation of Box C. [ ] See patent family annex.

*	Special categories of cited documents:	"T"	later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
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"P"	document published prior to the international filing date but later than the priority date claimed		

Date of the actual completion of the international search 05 MAY 1995	Date of mailing of the international search report 19 JUN 1995
Name and mailing address of the ISA/US Commissioner of Patents and Trademarks Box PCT Washington, D.C. 20231 Facsimile No. (703) 305-3230	Authorized officer DIEGO F.F. GUTIERREZ Telephone No. (703) 308-1113

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INTERNATIONAL SEARCH REPORT

International application No.
PCT/US95/02341

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US, A, 3,946,611 (LARSSON) 30 MARCH 1976.	
A	US, A, 4,195,055 (PATEL) 25 MARCH 1980.	
A	US, A, 4,195,057 (PATEL) 25 MARCH 1980.	
A	US, A, 3,243,303 (JOHNSON) 29 MARCH 1966.	
A	US, A, 3,479,877 (ALLEN ET AL.) 25 NOVEMBER 1969.	
A	US, A, 5,120,137 (OU-YANG) 09 JUNE 1992.	
A	US, A, 4,410,493 (JOSLYN) 18 OCTOBER 1983.	
A, P	US, A, 5,378,430 (NIEVES ET AL.) 03 JANUARY 1995.	

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US95/02341

Box I Observations where certain claims were found unsearchable (Continuation of item 1 of first sheet)

This international report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:

2. ☐ Claims Nos.:
because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:

3. ☒ Claims Nos.: 5 and 6
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

Box II Observations where unity of invention is lacking (Continuation of item 2 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

1. ☐ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims.

2. ☐ As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.

3. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:

4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest

☐ The additional search fees were accompanied by the applicant's protest.

☐ No protest accompanied the payment of additional search fees.